



Oxidation of Continuous Carbon Fibers Within a Silicon Carbide Matrix Under Stressed and Unstressed Conditions

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Prepared for the
24th Annual Conference on Composites, Advanced Ceramics, Materials, and Structures
sponsored by the American Ceramic Society
Cocoa Beach, Florida, January 23–28, 2000

National Aeronautics and
Space Administration

Glenn Research Center

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OXIDATION OF CONTINUOUS CARBON FIBERS WITHIN A SILICON CARBIDE MATRIX UNDER STRESSED AND UNSTRESSED CONDITIONS

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ABSTRACT

Carbon fiber reinforced silicon carbide matrix composites (C/SiC) are a ceramic matrix composite (CMC) material that offers benefits for use in a wide range of high temperature structural applications. However the susceptibility of the carbon fibers to degradation in oxidizing environments has hindered the material's use in certain applications requiring long lives under oxidizing conditions. The susceptibility of carbon fibers to oxidation will be discussed as well as the enhancement (improvement in oxidation resistance) of C/SiC materials. Thermogravimetric analysis of carbon fibers shows susceptibility to oxidation in two distinct kinetic regimes. However, in the thermogravimetric (wt. loss) analysis of unstressed, unenhanced, seal coated C/SiC coupons, the two regimes were not observed due to crack closure and matrix effects, which inhibited the oxidation process. Stressed oxidation (creep rupture) tests put the material under a stress, which is a more realistic condition for many applications. In stressed oxidation tests, the two oxidation kinetics regimes were observed. These tests can provide better insight into how the material will perform in applications involving stress. Stressed oxidation of enhanced materials containing oxidation inhibitors showed significantly improved lives at the specific test conditions considered, although there was susceptibility to oxidation at intermediate temperatures.

INTRODUCTION

Reinforcing carbon fiber within a silicon carbide matrix (C/SiC) is a type of ceramic matrix composite (CMC) that is currently undergoing considerable investigation for application in a wide range of aerospace applications. Some of the particular applications for which C/SiC is being investigated include thrusters, nozzles, nozzle ramps, gas generators, and turbopumps. This is because of the benefits offered by the material. It has more graceful failures compared to monolithic ceramics due to the reinforcement by the fibers. It also, in general, has a higher strength to density ratio and higher temperature capability than superalloys. The low density of C/SiC can reduce the overall weight of a launch vehicle so that benefits can be obtained in increased performance, lower launch or operation cost and/or increased payload. Another specific area that could be impacted would be in prolonging satellite life via more efficient use of propellants used for orbital maintenance. The increase in performance due to more efficient thrusters would translate into more efficient use of the propellants. In other applications, the use

of high-temperature components that require little or no cooling can allow for smaller and simpler engine designs which can have a beneficial effect in overall vehicle design so that weights and geometric complexities are reduced, performance is increased, and overall vehicle complexity is simplified.

The reinforcing carbon fibers within a silicon carbide matrix composite are susceptible to reaction and loss in oxidizing environments. This susceptibility of the fibers to oxidation has hindered C/SiC use in certain high-temperature, structural applications. To better understand the limitations of the material, environmental studies have been conducted on T300 carbon fiber tows and C/SiC composites. Once the kinetics of the oxidation process are understood, ways to protect the material through enhancements can be identified.

The susceptibility of carbon fiber to oxidation is dependent on many factors. The factors include activation energy of the carbon-oxygen reaction, impurities, crystal structure, carbon surface, environment, pressure, and temperature [1,2]. Many of these factors can vary greatly from one form of carbon to another. However, when dealing with only one type of carbon, environment and temperature are the only main variables. Other factors that must be considered have to do with when the fibers are in a matrix that has microcracks, which allow for the ingress of oxygen to the fibers. The cracks in C/SiC materials are present in the as-fabricated material. They form upon cooling down from the processing temperature due to stresses that arise from the difference in the coefficients of thermal expansion for the carbon fiber and the silicon carbide matrix. Factors that affect the oxidation of carbon in a matrix include overall crack density, crack size opening which is affected by thermal or mechanical strains, and enhancements that may seal cracks by forming solid oxides or glasses.

This paper will present the results of environmental exposure tests of C/SiC composites. First, the results of thermogravimetric analysis (TGA) tests will be presented for uncoated T300 carbon fibers and for C/SiC composites across a range of temperatures in a flowing oxygen environment. Then the results of stressed oxidation (creep rupture) tests in air will be presented. Finally, stressed oxidation results for two enhanced materials will be discussed and compared to two unenhanced materials.

EXPERIMENTAL

Thermogravimetric analysis was conducted on T300 carbon fiber. For each test run, a sample of approximately 0.4 grams of carbon fiber tow was placed in an alumina basket that was suspended in a furnace by a platinum wire. The test environment was oxygen flowing at a rate of 100 cc/min through a tube with a 1" inner diameter. With the sample already suspended in place, heat-up to the test temperature was started until the desired temperature was reached. Tests were conducted at temperatures of 600°C, 650°C, 700°C, 750°C, 800°C, 900°C, 1000°C, and 1100°C. Weight loss was monitored throughout the test. Tests were allowed to continue until complete carbon consumption occurred.

The ceramic matrix composite material used for the TGA testing and most of the stressed oxidation testing was DuPont-Lanxide Composites' (DLC, now known as Honeywell Advanced Composites, Inc.) standard C/SiC material. It had a continuous fiber 2-D woven (0/90) preform of carbon fiber in 1-k tows (bundles of 1000 individual fibers). The preform had a pyrolytic-carbon interphase that was applied through chemical vapor infiltration (CVI). The silicon carbide matrix was also applied through chemical vapor infiltration. Following machining of tensile bar coupons and TGA coupons, an external seal coating of silicon carbide was applied through chemical vapor deposition (CVD).

Thermogravimetric analysis was conducted on C/SiC composite coupons suspended in a furnace with oxygen flowing at a rate of 100 cc/min. The C/SiC coupon samples had dimensions of approximately 1.00"x0.50"x0.13". The as-received coupons had an external seal coating that was applied after machining of the edges and of a hole. The hole in the coupon allowed for it to be suspended in the furnace by a platinum wire. The temperatures of interest were 550°C, 600°C, 650°C, 700°C, 750°C, 800°C, 900°C, 950°C, 1000°C, 1100°C, 1250°C and 1400°C. Exposure was allowed to continue for a period of 25 hours.

Stressed oxidation tests were conducted on the C/SiC material. The tensile bar coupons were dog-bone shaped and had dimensions of approximately 6.00" long, 0.40" wide in the gage section, and 0.13" thick. The tensile bar coupons were loaded into an Instron 8500 Servo-Hydraulic load frame with hydraulic, water-cooled, wedge grips. A SiC susceptor coil placed circumferentially around the sample's gage region was used to heat the gage section of the test coupon. All instrumentation was placed within the gage section of the sample. Thermocouples placed at the front and rear face of the sample were used to monitor temperature. Extensometers with probes were placed on both edges of the coupon and were used to monitor strain. Samples were brought to the desired temperature. The test temperatures were 350°C, 550°C, 750°C, 1000°C, 1250°C, 1400°C, and 1500°C. Once at temperature, a tensile stress of either 69 MPa (10 ksi) or 172 MPa (25 ksi) was applied. Tests were conducted in an air environment. The tensile bar coupons remained under constant load at elevated temperature until they failed or until 25 hours had elapsed. Samples that survived 25 hours were fractured at room temperature to determine the residual strength. Stressed oxidation was also conducted on four different C/SiC materials at a temperature of 1454°C at stresses of 69 and 172 MPa in air. Descriptions of these four materials will be discussed in the Results section.

RESULTS

The results of the TGA tests of the bare carbon fiber show two different temperature regimes for carbon oxidation (see Figure 1). In regime 1, from 600°C-750°C, there is a strong temperature dependence. As temperature increases, the rate of carbon oxidation increases. However in regime 2, from 750°C-1100°C, there is not as strong of a temperature dependence. The rate of weight loss is similar for all temperatures in this second range. The transition from regime 1 to regime 2 is sometimes interpreted as a separate or third regime, often referred to as the transition regime.

The results of the TGA tests of the C/SiC composites in Figure 2 show very different trends than for the bare carbon fiber. At 550°C-700°C, the oxidation rate increased as temperature increased. The oxidation rate then remained nearly the same for temperatures of 750°C and 800°C. At these lower temperatures, complete fiber burnout occurred in the composites. The oxidation rate then began to decrease as temperatures approached 1100°C. At 1100°C, the oxidation rate was the lowest and there was only a 1% reduction in weight over the 25 hour period. Oxidation at other temperatures near 1100°C was also relatively minimal. Above 1100°C, at temperatures of 1250°C and 1400°C, the oxidation rate began to increase again. It should be noted that the coupons tested at 350°C and 750°C were from a different batch of material than the rest of the coupons. The fiber loading was lower in this material (approximately 43-wt % carbon fiber compared to approximately 50-wt % for the other batch of material).

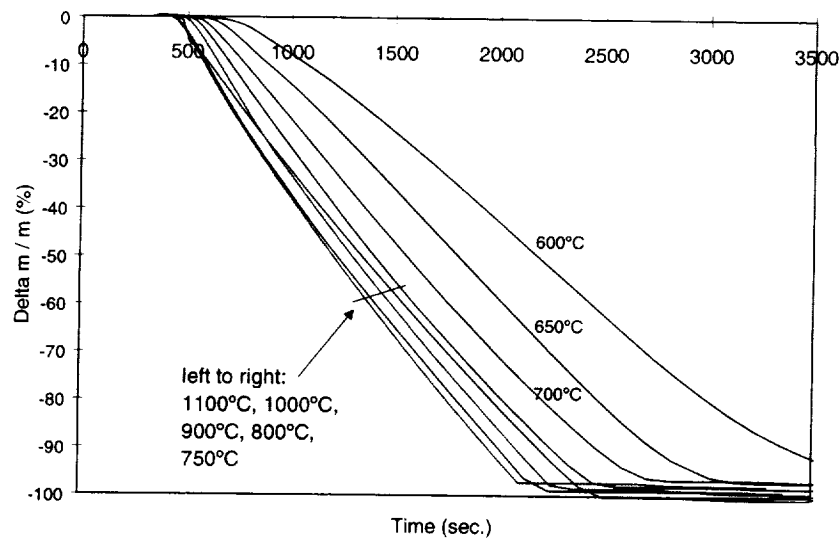


Figure 1. TGA results. Percent weight loss versus time for the oxidation of bare T300 carbon fiber.

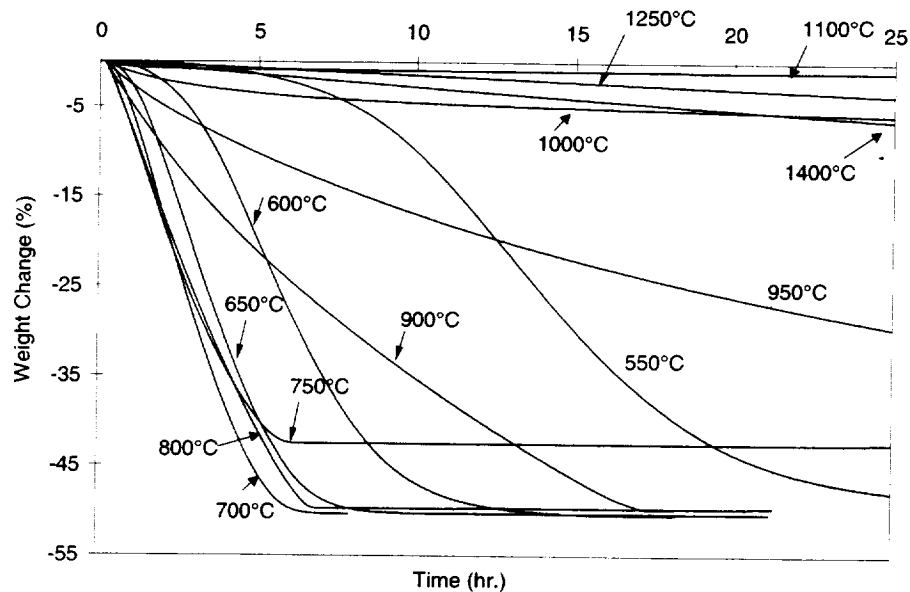


Figure 2. TGA results. Percent weight loss versus time for the oxidation of carbon fibers in DLC standard C/SiC composites.

The results of stressed oxidation testing in air are shown in Figure 3 and Table I. Times to failure for samples stressed oxidation tested at 172 MPa at temperatures ranging from 750°C-1500°C had lives of 17-36 minutes and fall to the left side of Figure 3. Times to failure at 69 MPa were longer in this same temperature range with lives of 65-142 minutes and fall to the right side of Figure 3. Changes in strain over time were observed. The strain is attributed to the effects of fiber and interphase oxidation, fiber failure, and load transfer rather than attributed to plastic deformation. However, three stages of creep similar to those that would occur during classical creep as seen in metals were observed at 69 MPa. For the 69 MPa tests, primary, secondary, and tertiary types of "creep" regimes were observed at all temperatures with the exception of the sample tested at 750°C which did not have a tertiary type of regime. The samples tested at 172 MPa did not exhibit such distinct regimes. Strains to failure seemed to

have more of a dependency on temperature rather than on load as indicated by the somewhat similar trends and values of strains to failure for 69 MPa and 172 MPa testing at a specific temperature. This is despite the higher stress being two and a half times greater than the lower stress. For both stresses there was an upward trend in strain to failure with increasing temperature. Sample lives, which were much longer at temperatures of 350°C and 550°C, are not shown in Figure 3 but appear in Table I. At 550°C, the sample tested at 172 MPa failed at the 25 hr point while the sample tested at 69 MPa survived 25 hr and had a residual strength of about one-half the ultimate tensile strength at room temperature. The samples tested at 350°C survived 25 hr of exposure under each stress condition. The residual strengths of these samples as determined at room temperature were within the range of strengths obtained for two as-received samples (351 and 406 MPa).

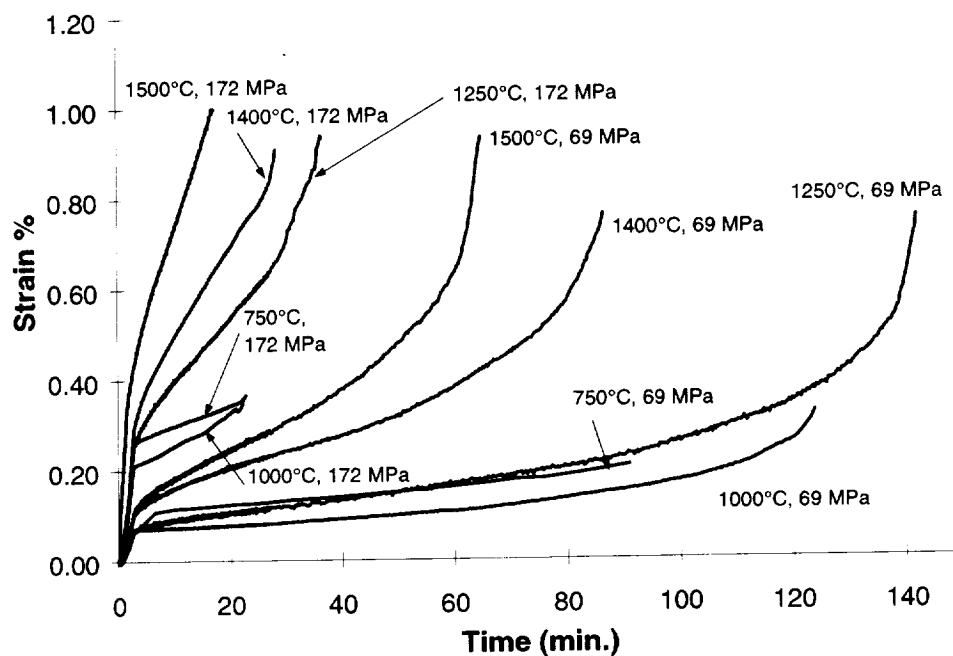


Figure 3. Stressed oxidation results. Strain versus time curves for the stressed oxidation of DLC standard C/SiC composites at stresses of 69 and 172 MPa.

Test Temp. °C	Time to Failure	Strain to Failure (%)	Residual Strength MPa
1500	65 min.	0.930	
1400	86 min.	0.757	
1250	142 min.	0.750	
1000	124 min.	0.324	
750	91 min.	0.209	
550	25+ hr.		192
350	25+ hr.		382

Test Temp. °C	Time to Failure	Strain to Failure (%)	Residual Strength MPa
1500	17 min.	1.000	
1400	28 min.	0.910	
1250	36 min.	0.944	
1000	22 min.	0.359	
750	22 min.	0.363	
550	25 hr.	0.450	
350	25+ hr.		403

Table I. Times and strains to failure during the stressed oxidation of C/SiC material. Data at 69 MPa is shown at the left. Data at 172 MPa is shown at the right.

Stressed oxidation was conducted on several different C/SiC materials at a temperature of 1454°C and stresses of 69 and 172 MPa. The four materials were DuPont Lanxide Composites' (DLC's) standard C/SiC material, DLC's enhanced C/SiC material, DLC's enhanced C/SiC material with a CBS external seal-coating, and a B.F. Goodrich C/SiC material. All materials had a [0/90] weave of 1k T300 carbon fiber tows with a pyrolytic-carbon interphase applied by CVI, a SiC matrix applied by isothermal CVI and an external seal-coating of SiC (C-B-Si for one of the materials) applied by CVD. The DLC standard C/SiC material is the same material as used in the previously mentioned TGA testing and stressed oxidation testing. The DLC enhanced C/SiC material had a yarn heat treatment and has a boron containing particulate in the composite. The DLC enhanced C/SiC material with a CBS coating has a boron containing particulate in the composite and an external seal coating that consists of carbon, boron, and silicon. It is believed that the BFG C/SiC did not have any oxidation inhibitors and should therefore have oxidation characteristics similar to the DLC standard C/SiC material.

The times to failure for the stressed oxidation of the four materials at a temperature of 1454°C and stresses of 69 and 172 MPa are shown in the bar graphs in Figure 4a and Figure 4b respectively. It can be seen that at 69 MPa both of the materials that were enhanced with boron performed better than the two materials without boron enhancements. The two boron containing materials ran out to 25 hours and had relatively high residual strengths as determined at room temperature. At a stress of 172 MPa, the DLC enhanced C/SiC did not perform as well. However the DLC C/SiC material with a CBS coating performed significantly better than the other three materials at this high stress and had lives in the low teens of hours. Under the program with NASA Marshall Space Flight Center under which these stressed oxidation tests were conducted at NASA Glenn Research Center, the ultimate tensile strengths (UTS) in air of two of the C/SiC materials at 1482°C (2700°F) were determined by the Southern Research Institute (SRI). The DLC standard C/SiC material had an UTS of 274 MPa and the DLC enhanced C/SiC material had an UTS of 307 MPa [3].

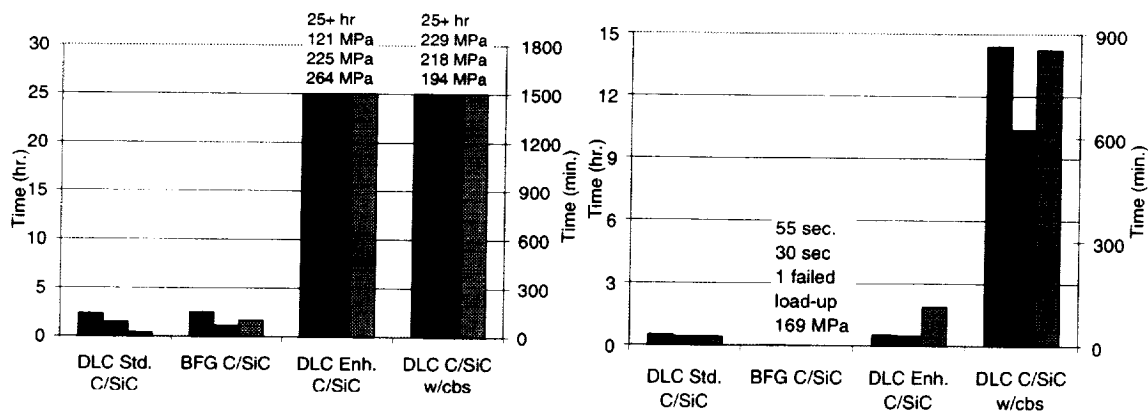


Figure 4a and 4b. Times to failure for four different C/SiC materials at a temperature of 1454°C and stresses of 69 MPa (left) and 172 MPa (right).

DISCUSSION

The results of the TGA tests for the bare unprotected carbon fiber show the susceptibility of carbon to oxidation in an oxygen environment for a wide range of temperatures. Oxidation rates in regime 1, which occurred at test temperatures from 600°C-750°C, showed a strong dependence on temperature. However in regime 2, which occurred at test temperatures from 750°C-1100°C, the reaction rates were not as strongly dependent on temperature.

The two regimes observed in the TGA test are explained by realizing that the recession or consumption of carbon (across a distance Δx [m]) is dependent on a two-step process occurring in series. The more limiting step controls the overall oxidation process. The first step is the diffusion or supply of oxygen, which is quantified by the diffusion coefficient (D [m^2/s]). The diffusion coefficient for oxygen diffusing in the product gases (CO or CO_2 from carbon and oxygen reactions) is not very strongly dependent on temperature and only increases in value by three times across the temperature range of 600°C-1500°C. The second step in the series is the rate of reaction between the oxygen and carbon, which is quantified by the reaction rate constant (K [m/s]). The reaction rate constant has an exponential dependence on temperature and can change by three orders of magnitude across this same temperature range.

The oxidation kinetics at a certain temperature can be predicted by considering a dimensionless parameter called the Sherwood number. The Sherwood number, Sh , is equal to the reaction rate constant times some characteristic distance, Δx [m], divided by the diffusion coefficient,

$$Sh = \frac{K \Delta x}{D}$$

The value of the Sherwood number directly correlates to the oxidation kinetics regime. At low temperatures, the reaction rate constant will have a very low value and the diffusion coefficient will also have a relatively low value but not as low as the reaction rate constant. For a given Δx value, the relatively low ratio of K/D [units of $1/\text{m}$] correlates to reaction controlled kinetics. At low temperatures there is a sufficient supply of oxygen, however the carbon/oxygen reactions are so slow that oxygen saturates the surrounding area. A low Sherwood value is therefore obtained at low temperatures and correlates to the reaction controlled kinetics regime.

At high temperatures, the value of the diffusion coefficient is at the high end of its range but the reaction rate constant has increased by several orders of magnitude. In this case (with the same Δx value), the relative ratio of K/D is very large. The carbon is very reactive with oxygen, and the carbon quickly consumes the oxygen. There will be a sharp gradient in oxygen concentration. The oxygen concentration will be high where it is first supplied, but can quickly decrease to zero at the carbon surface. At high temperatures, the slower step is the supply of oxygen so that the oxidation kinetics are gas phase diffusion controlled. At high temperatures, high Sherwood numbers are obtained.

Given the above discussion about kinetics and the results from oxidizing bare carbon fibers, some of the trends obtained for unstressed C/SiC coupons are not what would initially be expected, however the differences can be explained by matrix effects. As temperature was increased, the trends of the oxidation rate increasing due to a strong temperature effect (350°C-700°C) and of the oxidation rate showing less of a temperature dependence (700°C-800°C) were observed as for the bare fibers. However, at higher temperatures, the expected trend of similar high oxidation rates with little temperature effect was not seen. Instead,

the oxidation rates decreased as a certain temperature was approached (1100°C) then began to slightly increase again at higher temperatures. The decrease in oxidation rate at temperatures higher than 800°C can be explained by matrix effects. As temperature is increased toward the material's processing temperature (approximately 1100°C), the matrix cracks will begin to narrow as the thermal expansion of the matrix and fibers are matched. At the processing temperature, crack edges have come together and pinch off the supply of oxygen. Since crack edges may not align perfectly with one another, the fibers are not completely sealed off from the environment. However the carbon reaction rates can be significantly decreased as the cracks approach closure. Eckel et al. have shown that the parabolic reaction rate begins to be reduced once pores (crack openings) reach sizes below 1 micrometer and is greatly reduced once pore sizes of 10 nanometers are reached [2]. Another effect on the matrix cracks is the growth of a silica scale (SiO_2) as the SiC matrix oxidizes. The growth of the oxide scale can fill in cracks at temperatures sufficient for oxide growth and seal off the fibers from the outside environment. In the temperature range of 800°C -1100°C, the kinetics for the oxidation of SiC and the growth of SiO_2 are linear while for temperatures above 1100°C, the kinetics are parabolic [4]. The minimal weight loss in the C/SiC coupon at the temperature of 1100°C is at a temperature near the presumed processing temperature and is also in the parabolic growth regime for the SiO_2 scale. At higher temperatures, the oxidation increases and may be due to overexpansion of the SiC matrix relative to the carbon fibers. Compression of the crack edges can put a tensile stress on the fibers and cause a shift in the material so that crack edges become out of alignment and oxygen has a wider opening for ingress to the fibers.

In the TGA results, two kinetic regimes and the susceptibility of carbon to oxidation were observed for the bare carbon fibers while effects from matrix cracks and scale growth were observed for the unstressed C/SiC coupons. The stressed oxidation results for the unenhanced C/SiC coupons under a tensile load show that the presence of a stress greatly diminishes the matrix effects and allow oxidation regimes to be observed similar to those seen for the oxidation of bare carbon fiber. Analysis of the oxidation of carbon fibers in C/SiC CMCs under high stress conditions represents a more realistic or worst case scenario for how the material would perform in applications where the material is expected to be under stress. At temperatures from 350°C-750°C, the times to failure or residual strengths had a strong dependence on temperature. At temperatures higher than 750°C (1000°C-1500°C), the times to failure were relatively short. The small peak in times to failure at 1250°C may have to do with the processing temperature or matrix effects. Stressed oxidation results had two distinguishable regimes, similar to those observed for the oxidation of bare carbon fiber, and therefore also suggest two different rate controlling kinetics as carbon oxidizes.

The results of stressed oxidation testing of the four different C/SiC materials show how enhancements can significantly improve the life of the material. For the samples tested at 69 MPa, the DuPont Lanxide standard C/SiC and the B.F. Goodrich C/SiC had short lives that ranged from 24 minutes to 2.5 hr. Cross-sections taken from the heated gage section of these two materials showed significant oxidation of the carbon fibers. A shrinking core of carbon fibers was observed as oxidation occurred from the outer perimeter inward. This is an oxidation pattern that correlates to diffusion controlled kinetics, which is what would be expected at this high temperature. Failures of the two materials occurred in the heated gage region. In contrast, the DuPont-Lanxide materials that were enhanced with boron survived 25 hours and had favorable

residual strengths. Cross-sections taken from the hot zones showed very little oxidation. Both materials showed no significant evidence of oxidation of carbon interphases or fibers during analysis of polished cross-sections using an optical microscope. The boron enhancements appear to provide significant protection to the fibers at this lower stress.

At the higher stress of 172 MPa, the DLC standard C/SiC and the BFG C/SiC materials had very short lives. The DLC standard C/SiC material had lives of less than a half an hour. Cross-sections again showed a shrinking core for the oxidation of carbon fibers and carbon fiber tows. The BFG C/SiC material either failed upon load-up or had lives of less than a minute. Since lives were so short, less oxidation of the carbon fiber was observed however oxidation of portions of fiber tows from the edge inward was observed. The DLC enhanced C/SiC material at this higher stress had much shorter lives than at 69 MPa. The times to failure ranged from 0.5 hr to almost 2 hr. A cross-section prepared from one of the samples showed very minimal oxidation in the form of some oxidized interphases along a crack near the edge. Minimal oxidation in the hot-zone and failures in the thermal gradient region would suggest that the material failed at some intermediate temperature where the enhancement is not as effective. The DLC C/SiC material with a CBS coating had lives that were significantly longer than those of the other three materials. This material had lives in the low teens of hours. Very little evidence of oxidation in the cross-section taken from the gage section was observed. This material also failed in the thermal gradient region. Analysis of a section of material taken from a strip along the thermal gradient region showed oxidation of carbon fibers and interphases at a region low in the thermal gradient region. The longer lives in the material with boron in the matrix and in the external coating suggests that this enhancement approach provides better protection at intermediate temperatures than the material with boron only in the matrix. However, failures in the gage region for both materials suggests susceptibility to oxidation at low to intermediate temperatures where borosilicate glasses and silica scales do not form.

CONCLUSIONS

When carbon fiber without a matrix or interphase is allowed to react freely, oxidation will occur with a temperature dependence in either a reaction controlled regime or a diffusion controlled regime (Note that the transition between these two regimes is sometimes considered in literature as its own distinct regime, a third regime). The susceptibility of carbon fibers within a composite to degradation in an oxidizing environment is very dependent on whether or not a stress is present. In an unstressed matrix under oxidizing conditions, crack closure and matrix effects can inhibit oxidation at higher temperatures while there is still susceptibility at lower temperatures. This form of oxidation inhibition at higher temperatures can not be expected or relied upon when a stress is involved. When the material is under stress, significant crack closure and matrix effects are not seen and the two types of kinetics as noted for the bare fiber are observed. Comparison of the results of unstressed and stressed exposures show that unstressed C/SiC material at temperatures around 1100°C or 1250°C could suffer little weight loss due to minimal oxidation after 25 hours. However if the material is under a stress of 172 MPa, the material could fail after 28 minutes due to significant oxidation.

The use of enhancements was effective at inhibiting oxidation even when under stress. The enhancement methods had boron containing particulates in the matrix for one material and the second, in addition, had boron in the external seal coating. Both enhanced materials showed very significant increases in lives at 69 MPa. The materials survived 25 hrs of exposure and had favorable residual strengths. At the higher stress of 172 MPa, only the material with boron in

both the matrix and external seal coating showed an improvement in life, however, failures occurred in the thermal gradient region. Even though the material with boron only in the matrix failed in relatively short times at the higher stress, it did not show significant oxidation in its heated gage section. The enhancements in the two materials were beneficial at our specific conditions. However, the materials did show some susceptibility at intermediate temperatures as indicated by failures in the thermal gradient region and microstructural analysis.

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REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE July 2000		3. REPORT TYPE AND DATES COVERED Technical Memorandum
4. TITLE AND SUBTITLE Oxidation of Continuous Carbon Fibers Within a Silicon Carbide Matrix Under Stressed and Unstressed Conditions			5. FUNDING NUMBERS WU-523-61-13-00 1L161102AH45	
6. AUTHOR(S) Michael C. Halbig and Andrew J. Eckel				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) NASA Glenn Research Center Cleveland, Ohio 44135-3191 and U.S. Army Research Laboratory Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER E-12344	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001 and U.S. Army Research Laboratory Adelphi, Maryland 20783-1145			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-2000-210224 ARL-TR-2194	
11. SUPPLEMENTARY NOTES Prepared for the 24th Annual Conference on Composites, Advanced Ceramics, Materials, and Structures sponsored by the American Ceramic Society, Cocoa Beach, Florida, January 23-28, 2000. Michael C. Halbig, U.S. Army Research Laboratory, NASA Glenn Research Center; and Andrew J. Eckel, NASA Glenn Research Center. Responsible person, Michael C. Halbig, organization code 5130, (216) 433-2651.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Categories: 24 and 20 Distribution: Nonstandard This publication is available from the NASA Center for Aerospace Information, (301) 621-0390.			12b. DISTRIBUTION CODE	
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14. SUBJECT TERMS Ceramic matrix composites; Oxidation kinetics			15. NUMBER OF PAGES 16	
			16. PRICE CODE A03	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	

